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4. Sources of variability in single grain dose recovery experiments: insights from Moroccan and Australian samples

A methodological study on the characteristics of single quartz grains from different environmental contexts during OSL dose recovery experiments.

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Sources of variability in single grain dose recovery experiments: Insights from Moroccan and Australian samples

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Abstract

In our study, we investigate the quartz single-grain dose recovery characteristics of five aeolian samples from three archaeological sites in Australia and Morocco. Comparatively small (20 – 49 Gy) and high (180 – 208 Gy) doses were applied to sand-sized quartz grains of each sample. Samples were bleached by green laser, sunlight and solar simulator stimulation. We observed a primary dependency of the results on the size of the administered dose, but also observed sample-specific responses to the chosen dose recovery measurement parameters.

The Australian samples originate from an open-air archaeological site and consist of highly sensitized quartz grains with comparatively small equivalent doses. By contrast, the Moroccan samples originate from two cave sites known to be affected by heterogeneous dose rates and post-depositional mixing to varying degrees; this material is generally less sensitive, and expected equivalent doses are >180 Gy, while single-grain quartz weighted average signal saturation levels ($2D_0$) exceed 235 Gy.

Single grains from all sites, with one exception, recover small applied laboratory doses. These fall within 5 % of unity irrespective of the bleaching method. However, when applied doses are high, dose recovery test results vary substantially depending on how individual samples respond to the bleaching treatment prior to the given dose. The lowest dose recovery ratios and highest overdispersion values were observed in samples bleached in the solar simulator. Our results highlight the importance of investigating dose recovery characteristics at single grain level, and indicate additional sources of complexity in understanding the luminescence characteristics of quartz.

Keywords: OSL, single grain, quartz, SAR, dose recovery test, artificial bleaching, overdispersion

1. Introduction

In recent years, dating of single sand-sized grains of quartz using optically stimulated luminescence (OSL) has been frequently applied in geological and archaeological contexts. By reducing the aliquot size for equivalent dose (D_e) determination from multigrain aliquots to individual grain level, better resolution of dose distributions can be obtained.

The overdispersion value (σ_{OD}) allows quantification of the variability observed in single grain dose distributions (Galbraith et al., 1999), comprising both extrinsic (dose rate heterogeneity, incomplete bleaching and post depositional mixing) and intrinsic factors, such as counting statistics, instrument reproducibility, thermal transfer or other sample-specific OSL characteristics (Thomsen et al., 2007). Especially when dating material from highly complex settings such as cave sites, σ_{OD} of an individual sample can yield significant insight into, for example, the depositional history of sediments, thereby improving the reliability of age determinations (Jacobs et al., 2012).

Dose recovery experiments are one of the standard performance tests in quartz OSL dating, and are commonly assumed to represent a useful check of the suitability of measurement protocol parameters and the reliability of natural D_e estimates (Murray & Wintle, 2003).

In dose recovery experiments, known laboratory doses, which are recommended to be close to the expected natural D_e of a certain sample (Murray & Wintle, 2003), are administered either to artificially bleached or modern analogous samples. Their ability to recover this given dose within acceptable ranges – calculated as a ratio normalized to the applied dose – is assessed. Thomsen et al. (2012) have shown that the size of the administered dose is of considerable importance in dose recovery tests for single grains as well as for multiple grain aliquots. They found that, their quartz samples recovered relatively small given doses within unity, but at higher administered doses of 103 and 208 Gy, dose recovery was substantially underestimated by 10 – 15 %. As dose recovery tests are by definition laboratory-based experiments, the observed σ_{OD} in resulting single grain dose distributions only reflects the intrinsic variability in the individual sample and is not influenced by extrinsic factors, as is the case for natural samples (Thomsen et al., 2007). Furthermore, Guérin et al. (2015) recently found no correlation between dose recovery ratios and accuracy for obtained ages in 19 single-grain samples with independent age control. These observations cast doubt on the standard dose recovery test as a check for sample reliability, and highlight a need to investigate dose recovery tests in greater depth.

Given the complex nature of many cave-based archaeological sediments, dose recovery tests are often undertaken by resetting the natural signal rather than using modern analogues. Resetting can be achieved by various light sources which simulate natural bleaching conditions. The most commonly used bleaching sources are natural sunlight, solar simulators (SOL2), and stimulation in the OSL reader by green lasers or blue light emitting diodes (LEDs) (e.g. Aitken & Smith, 1988; Ballarini et al., 2007; Kang et al., 2012; Li & Wintle, 1991, 1992). Solar simulators (SOL2) have recently been shown to cause inaccuracies in dose recovery tests on multigrain aliquots (Choi et al., 2009; Wang et al., 2011). Thomsen et al. (2016) on the other hand, observed no significant systematic differences in dose recovery test results after SOL2 and blue LED bleaching for both, single grains and multigrain aliquots.

As yet it is unclear whether experimental dose recovery tests using artificial bleaching sources can accurately recover dose for all samples, especially in single grains, since generally dose recovery tests are undertaken on multigrain aliquots – even in a number of single grain dating studies (e.g. Arnold & Demuro, 2015; Demuro et al., 2012; Jacobs et al., 2008).

In this study we undertook single grain dose recovery tests (e.g. natural D_es, signal intensities) on five sand-sized quartz samples from three different environmental settings from archaeological sites in Australia and Morocco. We investigate the dose recovery characteristics of these samples following different bleaching methods and after administering doses of different magnitude. We discuss the potential impact of experimental design and sample luminescence properties on the obtained results.

2. Instrumentation and sample characteristics

2.1. Instrumentation

All OSL measurements were undertaken using an automated Risø TL-DA-20 reader with a single grain attachment (Bøtter-Jensen et al., 2003), equipped with a calibrated $^{90}Sr/^{90}Y$ beta irradiation source (Bøtter-Jensen et al., 2000), and fitted with a 7.5 nm Hoya U-340 filter (Bøtter-Jensen, 1997). Individual grains were mounted on single grain sample discs containing 100 holes (300 µm diameter) on a 10 x 10 grid. Optical stimulation was provided by 10 mW 532 nm solid-state green laser beams for 1 s at 125 °C (90 % power, ~50 W/cm^2 power density (Duller et al., 1999) and infrared LEDs (875 nm wavelength, ~130 mW/cm^2 power density).

2.2. Samples

We investigated five samples from archaeological sites that were deposited by eolian processes. We used quartz of the 180 – 212 µm sand-size fraction. Samples were extracted by a combination of wet and dry sieving, followed by chemical treatment (HCl, H_2O_2, density separation and HF) under subdued red light.

Three samples originate from two archaeological cave sites in Morocco. L-EVA-1083 was collected at the inland site of Rhafas (Doerschner et al., in revision; Mercier et al., 2007; Wengler, 1993), and L-EVA-1218 and L-EVA-1221 from Thomas Quarry I (TQ I), located in aeolianite in Casablanca city on the Atlantic coast (Rhodes et al., 2006). Both sites are caves filled with Pleistocene sediments that were affected by post-depositional carbonate cementation and dose rate heterogeneity of varying degrees. The proportions of individual grains emitting detectable luminescence signals for natural D_e determination are 23 – 66 % for Rhafas and 6.8 – 31 % for TQ I samples (proportions reflect full suite of samples collected at Rhafas and TQ I). Furthermore, these samples yield particularly high D_0 values which allow reliable determination of natural D_e between 126 and 216 Gy (using the Central Age Model (CAM) proposed by Galbraith et al., 1999), with σ_{OD} reaching up to 47 % (Table 1).

We compare these samples with two dune samples from the open-air archaeological site at Lake Mungo (LM), Australia (L-EVA-1010 and -1012; Fitzsimmons et al., 2014). These two samples yield CAM-derived natural D_e values of 42 and 21 Gy respectively, and σ_{OD} of less than 25 % (Table 1). Although previous work has already demonstrated that these samples exhibit bright, rapidly decaying OSL signals typical of highly sensitive quartz and are therefore well suited to OSL dating (Fitzsimmons, 2011; Fitzsimmons et al., 2014), it has also been reported that variations in microdosimetry in sediments from this region might induce relatively high σ_{OD} values in natural D_e distributions (Lomax et al., 2007).

To examine the different sensitivity characteristics of the individual samples, the sensitivity of the first test dose response T_N (in counts/seconds/Gy) of each single grain, obtained after measurement of the natural signal, was plotted

Table 1. Results of natural D_e determination and dose recovery tests.

Sample	Natural equivalent dose				Given	Test	Dose recovery green laser				Dose recovery sunbleached				Dose recovery SOL2			
	n^1 (%)	$D_e{}^2$ (Gy)	$\sigma_{OD}{}^2$ (%)	$D_0{}^3$ (Gy)	dose (Gy)	dose (Gy)	n^1 (%)	recovery ratio^2	$\sigma_{OD}{}^2$ (%)	$D_0{}^3$ ((Gy)	n^1 (%)	recovery ratio^2	$\sigma_{OD}{}^2$ (%)	$D_0{}^3$ ((Gy)	n^1 (%)	recovery ratio^2	$\sigma_{OD}{}^2$ (%)	$D_0{}^3$ ((Gy)
Lake Mungo																		
L-EVA-1010	16.7	42±1	24±2	65±3	49	10	24.0	0.95±0.01	7±1	67±3	18.2	0.96±0.01	6±1	65±4	18.7	0.95±0.01	10±1	63±6
					185	36	16.7	0.80±0.02	18±2	115±5	14.0	0.79±0.01	12±1	116±7	12.2	0.81±0.02	14±2	118±5
L-EVA-1012	9.0	21±1	24±3	73±9	20	4	21.5	0.96±0.01	6±1	72±3	18.5	0.97±0.01	9±1	53±3	10.1	0.94±0.01	11±1	63±7
					185	36	17.8	0.67±0.02	25±2	104±4	9.8	0.86±0.02	12±2	131±8	12.7	0.68±0.03	35±3	111±5
Rhafas																		
L-EVA-1083	4.1	216±12	36±4	208±16	20	4	11.2	0.97±0.01	9±1	51±5	6.3	0.83±0.02	22±2	76±11	6.5	0.99±0.02	10±2	59±7
					208	42	7.0	0.94±0.02	9±2	167±13	5.0	0.87±0.02	6±2	161±8	7.2	0.75±0.03	25±3	148±6
Thomas Quarry I																		
L-EVA-1218	3.9	126±9	47±5	118±7	20	4	16.7	0.93±0.01	5±1	47±3	4.1	0.91±0.02	8±2	40±3	7.5	1.04±0.02	7±2	75±5
					180	36	4.3	0.89±0.03	19±3	132±7	5.5	0.85±0.02	7±3	130±6	6.4	0.75±0.03	27±3	116±8
L-EVA-1221	4.4	153±7	34±4	149±6	20	4	16.8	0.95±0.01	6±1	49±3	4.3	0.93±0.02	9±2	59±6	11.2	0.97±0.01	9±1	59±5
					205	42	7.0	0.81±0.02	15±3	132±5	7.1	0.78±0.02	15±2	138±6	7.3	0.76±0.02	19±2	148±9

[1] Percentage of accepted grains.

[2] Determined using the Central Age Model (Galbraith et al., 1999).

[3] Weighted average D_0 value of the accepted D_es and standard error of the mean.

Table 2. Single grain characteristics of dose recovery experiments.

Sample	Given dose (Gy)	n^1	detectable signal^2 (100 %)	accepted grains (%)	No L_N/T_N intersection (%)	Remaining grains (100 %)	Dim grains^3 (%)	Recuperation > 5 % (%)	Recycling ratio (> 20 %) (%)	Depletion by IR (%)	D_e error > 30 % (%)	Grubbs test^4 (%)	$D_e > 2D0$ (%)
Lake Mungo													
L-EVA-1010	49	1800	1483 (82 %)	24.6	3.1	1072	17.0	0.8	57.8	19.5	4.3	0.1	0.6
	185	1800	1494 (83 %)	17.1	18.4	963	13.6	2.8	33.2	17.6	22.8	0.3	9.7
L-EVA-1012	20	2100	1504 (72 %)	22.2	0.2	1170	24.2	0.9	54.4	19.9	0.6	0.1	0.0
	185	2000	1525 (76 %)	17.5	18.9	972	18.6	4.4	31.0	19.7	18.3	0.1	7.8
Rhafas													
L-EVA-1083	20	2900	1447 (50 %)	14.7	0.3	1230	34.3	2.0	47.0	14.4	2.1	0.2	0.0
	208	2500	1343 (54 %)	11.9	14.2	990	22.3	22.1	34.1	9.6	7.5	0.2	4.1
Thomas Quarry I													
L-EVA-1218	20	2500	1451 (58 %)	14.1	0.3	1242	44.1	0.0	42.0	11.8	1.6	0.5	0.0
	180	2800	1884 (67 %)	7.9	25.8	1241	27.4	1.0	45.1	8.8	14.3	0.0	3.3
L-EVA-1221	20	2400	1530 (64 %)	14.8	0.4	1303	38.2	0.2	47.7	12.0	1.5	0.4	0.0
	205	3000	2309 (77 %)	9.3	23.3	1492	24.2	1.4	46.2	10.5	13.2	0.1	4.2

[1] Total number of grains measured per sample.

[2] Percentage of grains yielding an initial luminescence signal above background, and for which interpolation of the sensitivity-corrected natural signal using a single saturating exponential dose response curve resulted in finite dose estimates.

[3] Percentage of grains rejected due to insufficient test-dose signal.

[4] Percentage of grains identified as statistical outliers (Grubbs, 1950).

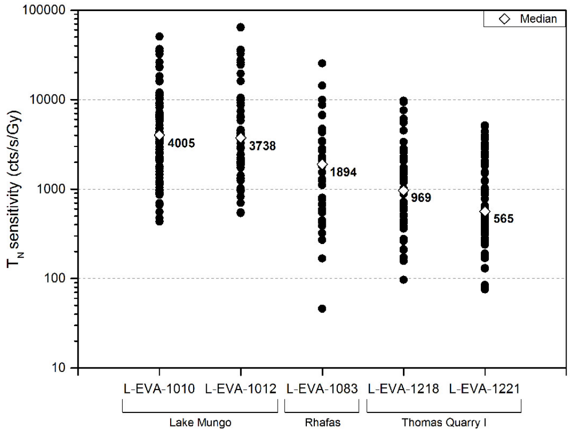

Figure 1. Luminescence sensitivity of single grains calculated from the OSL signal arising from the test dose immediately following natural D_e measurement (T_N) for all samples in this study (note logarithmic scale). Median values are indicated as open diamonds. Individual background corrected T_N signals were multiplied by 28.75 (1/0.035, integration in seconds) and then divided by the given test dose (Table S1) for comparability between samples.

(Fig. 1). Only individual grains accepted for natural D_e determination were incorporated to this figure; the test doses as applied to the samples using the single-aliquot regenerative-dose (SAR) protocol are listed in Table S1. The Australian samples yield high proportions of very bright grains ($> 10^4$ cts/s/Gy) and few grains emitting signal intensities of less than 10^3 cts/s/Gy. The samples from TQ I consistently yield single grain signal intensities in the range of $10^2 – 10^4$ cts/s/Gy. The sample L-EVA-1083 from Rhafas exhibits the most variable grain sensitivity; its calculated median sensitivity value is half that observed for the Australian samples.

In order to visualize the dominant OSL signal components in the samples, we undertook linearly-modulated OSL (LM-OSL) measurements on small (1 mm) aliquots at 125 °C using LEDs (470 nm, $\sim$40 mW/cm^2), following the procedure described by Bulur (1996). After preheating at 260 °C for 10 s, light intensity was increased from 0 to 90 % power over 500 s. The LM-OSL curves illustrate that all samples are dominated by the fast OSL signal component (Fig. S1). Single grain decay and growth curves of sample L-EVA-1221 (TQ I) and L-EVA-1010 (LM) also show that the samples are characterised by individual quartz grains with bright luminescence signals (Fig. 2). Weighted average D_0 values are higher in the Moroccan than the Australian samples. The determined D_e values – obtained using single saturating exponential curve fitting of the single grain dose response curves – for all samples in this study lie well below signal saturation levels (Table 1).

3. Dose recovery test – experimental details

Dose recovery tests on single grains were performed using the standard SAR protocol (Murray & Wintle, 2000, 2003) with a preheat temperature of 260 °C for 10 s, and a cutheat temperature of 220 °C. Preheat temperatures were determined based on the results of standard preheat plateau tests as well as combined dose recovery preheat plateau tests, in which seven different preheats (160 – 280 °C, data not shown) were applied to 1 mm multigrain aliquots (Murray & Wintle, 2003; Wintle & Murray, 2006). Potential feldspar contamination was tested at the end of each protocol by measuring the IR depletion of the OSL signal (Duller, 2003).

OSL signals were summed over the first 0.035 s of stimulation and corrected for background using the subsequent 0.035 s (Ballarini et al., 2007; Cunningham & Wallinga, 2009). Laboratory dose response curves were fitted using a single saturating exponential passing through the origin. Single grains were accepted for final analyses only when interpolation of the sensitivity-corrected natural signal on the dose response curve: 1) resulted in a finite dose estimate; 2) uncertainty on the natural test dose response was less than 20 % (test doses are given in Table 1); 3) were not affected by equivalent dose error > 30 %; and 4) passed the recuperation- (< 5 %), recycling- (< 20 %) and IR-depletion ratio tests (< 5 %). In addition, grains were rejected when exhibiting D_e signals exceeded saturation level (2 D_0) as suggested by Wintle & Murray (2006). Average dose recovery test ratios were calculated using the CAM.

Dose recovery tests were undertaken for each sample by applying two different laboratory doses and three different types of light exposure for bleaching. One applied dose was chosen to be close to the natural D_e (Murray & Wintle, 2003) and varied – depending on the sample – between 20 and 208 Gy (Table 1). A second set of dose recovery tests were performed for comparison using a laboratory dose of 185 Gy for the Australian samples (L-EVA-1010 and -1012) and 20 Gy for the Moroccan samples (L-EVA-1083, -1218 and -1221). Single grains from each sample were bleached by 1) the green laser (1 s at room temperature) in the OSL reader, 2) sunlight on the window sill (> 7 days, behind glass and therefore not entirely analogous to natural bleaching conditions), and 3) SOL2 at a lamp/sample distance of 60 cm. Stimulation in the Hönle solar simulator (equipped with a Hönle H2 filter, transmission range > 295 nm) by SOL2 was performed for 5 min to make sure that the individual grains were completely bleached while also avoiding underestimation of the recovered dose, as has been demonstrated for exposure times exceeding 1 hour (Choi et al., 2009). The total amount of individual grains measured for each dose recovery experiment is listed in Table S1.

4. Results

4.1. Acceptance and rejection of individual grains

Detailed information about the rejection of single grains for the different dose recovery experiments is listed in Ta-

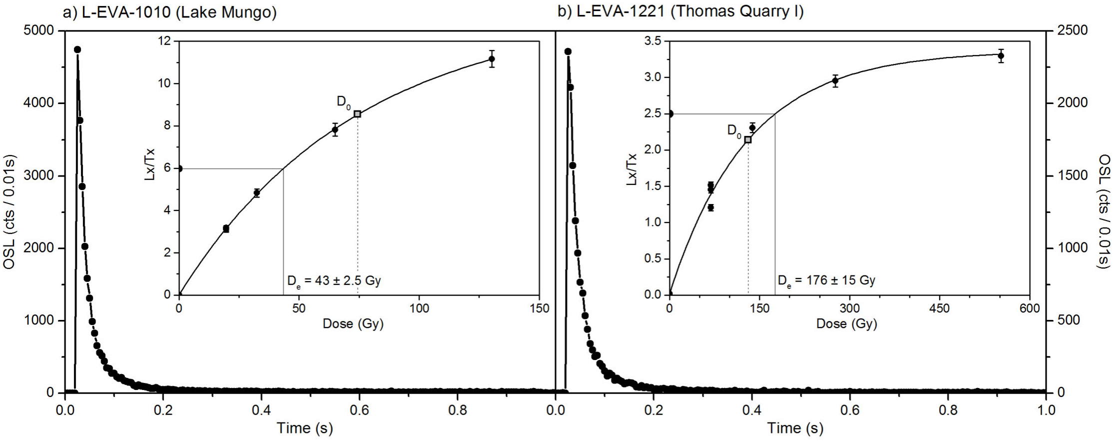

Figure 2. Natural OSL single grain decay and (as inset) dose-response curves for samples (a) L-EVA-1010 and (b) L-EVA-1221.

ble 2. Australian samples are more likely to give detectable luminescence signals for the chosen test doses (Table 1) than the Moroccan samples (LM 72 – 83 %, Rhafas 50 – 54 %, TQ I: 58 – 77 %, Table 2). This arises from the variability in intrinsic luminescence properties; the Australian samples generally comprise higher proportions of bright grains than the Moroccan samples (Fig. 1). Out of the grains exhibiting detectable luminescence signals, 14 to 26 % were rejected due to oversaturation (grains showing a L_n/T_n ratio well above maximum L_x/T_x value of the generated dose response curve) when administered doses are high (180 – 208 Gy), while this is only the case for 0.3 – 3.1 % of grains when administered doses are small (20 – 49 Gy). Sample L-EVA-1083 (Rhafas) yields the lowest rejection rate due to oversaturation at high given doses. This is most likely a consequence of its high signal saturation level after administration of a high dose, revealing weighted average D_0 values > 148 Gy in our dose recovery experiments (Table 1).

D_0 values calculated for all samples are consistent between bleaching types (Table 1). They do, however, show large variability depending on the given dose, with D_0 values arising from low administered doses yielding significantly smaller values than those arising from high doses. This is most likely caused by the different regeneration doses of the dose recovery measurements (Table S2). Maximum regeneration doses are considerably smaller after low given doses in comparison with high given doses. These results indicate that D_0 can presumably only be accurately determined when dose response curves are taken up to large doses.

Oversaturated grains were excluded from further analyses of rejection criteria in Table 2 to avoid statistical bias between dose recovery experiments with high and low given doses. As previously mentioned, regeneration doses vary in dose recovery experiments depending on the size of the given dose (Table S2). Therefore, observed differences in criteria causing rejection of individual grains between low and high given doses might partly be caused by those different measurement parameters. Depending on the sample site, individual grains are more likely to be rejected after a low administered dose due to poor recycling ratios (LM), insufficient test dose signals (TQ I) or a combination of both (Rhafas). While TQ I samples at high given doses only show a significant increase in rejection rates due to D_e errors exceeding 30 %, samples from LM are additionally affected by single grains failing the $2D_0$ criterion. Single grains from Rhafas increasingly fail due to recuperation values of > 5 %.

The number of individual grains passing all rejection criteria for dose determination varies considerably between sampling locations (LM: 10 – 24 %, Rhafas: 5 – 11 % and TQ I: 4 – 17 %; Table 1, Fig. 3a). Although a large number of single grains were rejected due to oversaturation solely when given doses were high, there is no clear correlation – with the exception of sample L-EVA-1010 (LM) – between the proportion of accepted grains and the size of the given dose. It is interesting to note that exceptionally high acceptance rates (11 – 24 %) were achieved for all samples when low doses were applied following green laser bleaching (Fig. 3a).

4.2. Differences observed in dose recovery ratios

Figure 3b summarizes the results of the measured/given ratios for each sample in the dose recovery experiment. The Australian samples from LM (L-EVA-1010 and -1012) and sample L-EVA-1221 from TQ I (Morocco) yield dose recovery ratios within 7 % of unity after low given doses, regardless of bleaching type (Fig. 3b, Table 1). By contrast, when given doses are high, these samples underestimate the measured/given ratios by 14 – 33 %. For the remaining Moroccan samples from Rhafas (L-EVA-1083) and TQ I (L-EVA-1218), recovery ratios vary between 1 – 17 % and 6 – 25 % of unity following low and high applied doses, respectively.

The type of bleaching source appears to influence dose recovery ratios for the Moroccan samples (Rhafas and TQ I). SOL2 stimulation at small given doses consistently results in values with the lowest (1 – 4 %) deviation from unity. Bleaching by sunlight results in the highest (7 – 17 %) devi-

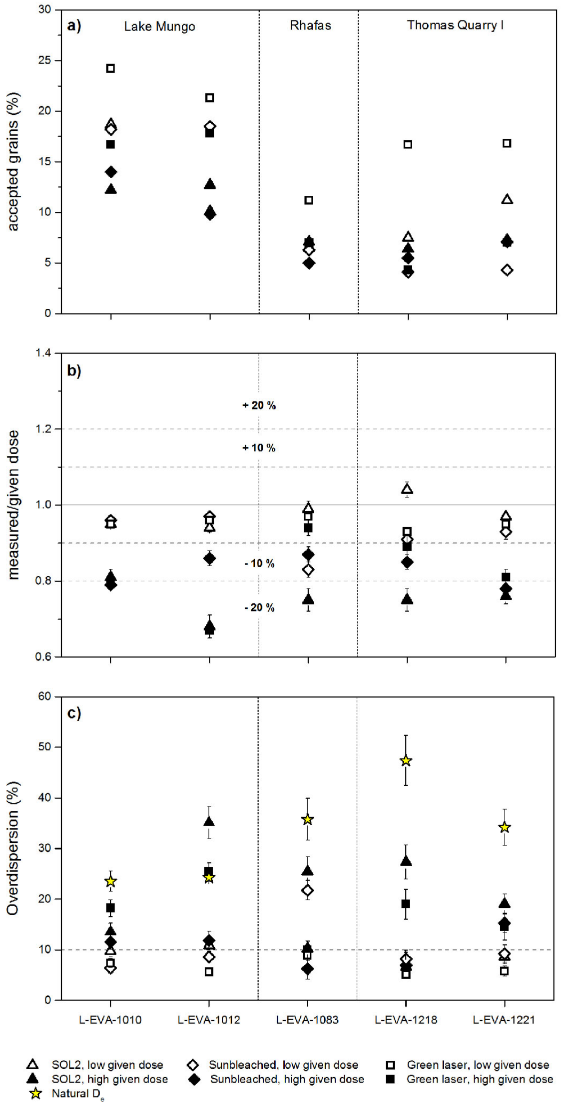

Figure 3. Results of single grain dose recovery experiments: (a) number of accepted grains after applying rejection criteria, (b) measured/given dose ratios and (c) calculated σ_{OD} values. Individual grains from each sample were bleached with green laser (squares), natural sunlight (diamonds) and SOL2 (triangles) prior to administering low (open symbols) or high (filled symbols) doses. Overdispersion values determined for the natural D_es are indicated by yellow stars.

ation of dose recovery from unity. Conversely, when given doses are high, green laser bleaching results in the most accurate (6 – 19 %) and SOL2 the least accurate (25 – 26 %) measured/given ratios. The Australian sample L-EVA-1012 (LM), however, yields consistent values of underestimation after high given doses for both, SOL2 and green laser bleaching (32 – 33 %).

To examine potential correlation between the degree of grain sensitivity and the accuracy of the dose recovery test results, measured/given ratios were plotted as a function of the corresponding single grain L_N signal intensities (Fig. S2). Individual grains from all samples emitting high intensity signals yield measured/given ratios close to unity when given doses are small. This characteristic decreases with decreasing signal intensity (Fig. S2a-e). The shape of the dose distributions, however, varies between samples. Individual grains from Rhafas (L-EVA-1083, Fig. S2c) are more widely scattered than LM or TQ I samples, whose distributions are relatively narrow. These results indicate a stronger correlation between signal intensity and accuracy of the dose recovery ratio. Similar single grain distributions following dose recovery experiments have also been observed in other studies (Duller et al., 2000; Jacobs et al., 2006; Thomsen et al., 2007). By contrast, when given doses are high, samples from all three sites exhibit a different behaviour. Single grains inconsistently over- and under-estimate the measured/given ratios, independently of L_N signal intensity (Fig. S2f-j).

The insets of Figure S2 show a correlation between measurement precision and L_N signal intensities for all samples. This is expected, as the impact of counting statistics decreases with increasing signal brightness (Duller et al., 2000).

4.3. Overdispersion variability in recovered doses

It has been shown that even under the controlled laboratory conditions of dose recovery tests, whereby all extrinsic factors can be excluded, σ_{OD} values of 7 – 12 % are nevertheless observed in single grain dose distributions (Jacobs et al., 2006; Reimann et al., 2012; Thomsen et al., 2005). This intrinsic σ_{OD} is caused by luminescence characteristics inherent within sand grains from a sample.

We calculated the σ_{OD} values for each single grain dose recovery test. The results, together with the σ_{OD} values from the natural D_es, are plotted in Figure 3c (see also Table 1). The behaviour of the Australian material (LM) and sample L-EVA-1221 (TQ I) differs from the other Moroccan samples by consistently revealing intrinsic σ_{OD} values of ∼10 % when administered doses are small, and substantially increased σ_{OD} values of up to 35 % following larger given doses.

For samples L-EVA-1083 (Rhafas) and L-EVA-1218 (TQ I), σ_{OD} values of 5-10 % (Table 1) are observed for almost all dose recovery test parameters. This is consistent with intrinsic σ_{OD} values reported in previous studies. A remarkable increase in σ_{OD}, however, can be observed for L-EVA-1083 when a small dose was given after sunlight bleaching (22 %), for L-EVA-1218 when a high administered dose was combined with green laser stimulation (19 %), and for both samples when high given doses and SOL2 were used (25 – 27 %).

Applying SOL2 stimulation prior to a high given dose results in the highest σ_{OD} (19 – 35 %) in all samples, except L-EVA-1010 (LM). For the sake of completeness, it should be noted that for sample L-EVA-1012 (LM) the intrinsic σ_{OD} following green laser (25 %) and SOL2 bleaching (35 %) at high given doses exceeds the value determined from the natural D_e (24 %, Fig. 3c). Those values are, however, not entirely comparable, since the given dose (185 Gy) is considerably higher than the natural D_e estimate (∼21 Gy) and, therefore, intersection of the sensitivity-corrected natural signal of the individual grains occurs along different parts of the dose response curves. Intrinsic σ_{OD} after sunlight stimulation of the material prior to a high given dose (12 %) is entirely comparable to the σ_{OD} obtained for all bleaching treatments and small administered doses (6 – 11 %).

Given the highly variable results of our dose recovery experiments, we cannot confidently specify the proportion of intrinsic σ_{OD} within the natural D_e σ_{OD} for most samples. Comparisons of the internal σ_{OD} from the dose recovery experiments close to the expected natural D_es with the measured σ_{OD} from the natural D_es of each sample indicate that considerable proportions of the σ_{OD} value are caused by extrinsic factors. As neither post depositional mixing, nor incomplete bleaching is likely for any of our samples (Doerschner et al., in revision; Fitzsimmons et al., 2014; Rhodes et al., 2006), high extrinsic σ_{OD} most likely results from heterogeneous dose rates (Lomax et al., 2007). Autoradiography was undertaken at the University of Bern to verify this assumption by highlighting spatially resolved radiation inhomogeneities in our samples following the procedure described in Rufer & Preusser (2009). Autoradiographs are shown in Figure S3; high-radiation emitters are visible as black hotspots and indicate dose rate heterogeneity, thereby explaining the high values obtained for external σ_{OD}. Furthermore, visual inspection under a microscope indicates that carbonate shielding of quartz grains, which could introduce further variation in microdosimetry by reducing the dose rate received by individual grains (Olley et al., 1997), might additionally affect L-EVA-1083 (Rhafas). This effect is, however, absent from LM and TQ I samples.

5. Discussion

5.1. Dose recovery and overdispersion

In this study the dose recovery characteristics of single quartz grains of archaeological samples from two Moroccan (Rhafas and TQ I) and one Australian (LM) site were examined.

The Australian samples consist of highly sensitized, bright quartz grains which are able to recover a small laboratory given dose (close to their natural D_e) within 6 % of unity and homogeneously (σ_{OD} 6 – 11 %) over a large number of individual grains. By contrast, when administered doses are high (185 Gy), the total number of single grains passing rejection criteria decreases (Table 1), while σ_{OD} increases (12 – 35 %) and the measured/given dose ratio is systematically underestimated by up to 33 %.

For the Moroccan samples, the results are variable. The samples originate from two cave sites and consist of individual quartz grains significantly less bright than the Australian samples. These results suggest that the Moroccan material underwent comparatively fewer sensitization cycles (cycles of dose and light exposure) than the Australian samples (Moska & Murray, 2006; Pietsch et al., 2008). Dose recovery ratios and σ_{OD} values in sample L-EVA-1083 (Rhafas) and L-EVA-1218 (TQ I) are highly variable (Fig. 3). Single grains from these samples recover low administered doses with similar accuracy (1 – 17 %) and precision (σ_{OD} 5 – 22 %) as high doses (within 6 – 25 % of unity, σ_{OD} 6-27 %). They also do not show significant dependency on the bleaching treatment. Therefore, it seems most likely that the results reflect a sample-specific response to the chosen dose recovery test parameters (bleaching type and size of administered dose).

For sample L-EVA-1221 (TQ I), however, measured/ given dose ratios and σ_{OD} values are clearly dependent on the magnitude of the given dose. Consequently, our results suggest that the SAR protocol would have to be declared as unsuitable for dating sample L-EVA-1221, in the case where a standard dose recovery test with a given dose close to the expected natural D_e (205 Gy) is applied. L-EVA-1221, however, passes a SAR suitability check when applied doses are low (20 Gy). Interestingly, L-EVA-1218 (TQ I) and L-EVA-1083 (Rhafas) pass standard SAR suitability checks when green laser bleaching is applied, yet fail after sunlight or SOL2 treatment, when given doses are close to their expected natural D_e (180 and 208 Gy, respectively).

Our contradictory results raise questions as to the validity of dose recovery tests in general, and more particularly as to the adequate choice of test parameters to interrogate the suitability of the SAR protocol. The Moroccan material is in general less sensitive than the Australian samples. While single grain signal intensity in all samples correlates positively with accuracy of measured/given ratio when given doses are small, even bright grains are not necessarily able to reproduce a given dose > 180 Gy with any accuracy. The observed variability in intrinsic σ_{OD} in our samples is alarming, particularly when considering that the correct assessment of natural σ_{OD} is critical for reliable age determination. Underestimation of the intrinsic, and thereby overestimation of the extrinsic, σ_{OD} might result in misinterpretation of single grain dose distributions and/or selection of inappropriate statistical age models. Autoradiographs suggest that increased values of extrinsic σ_{OD} in all samples result from variations in their microdosimetry.

5.2. Single grain rejection criteria

Application of single grain rejection criteria significantly reduces the amount of acceptable grains in each dose recovery experiment presented in this study. The justification of single grain standard rejection criteria (recycling, IR depletion and recuperation) has been recently questioned in several studies (e.g. Geach et al. 2015; Guérin et al. 2015; Thomsen et al. 2012; Zhao et al. 2015. Thomsen et al. (2016) reported that standard rejection criteria for single grains "do not result in significant changes in either dose or overdispersion of the single grain distributions" in the samples that they investigated. To investigate the impact of the chosen single

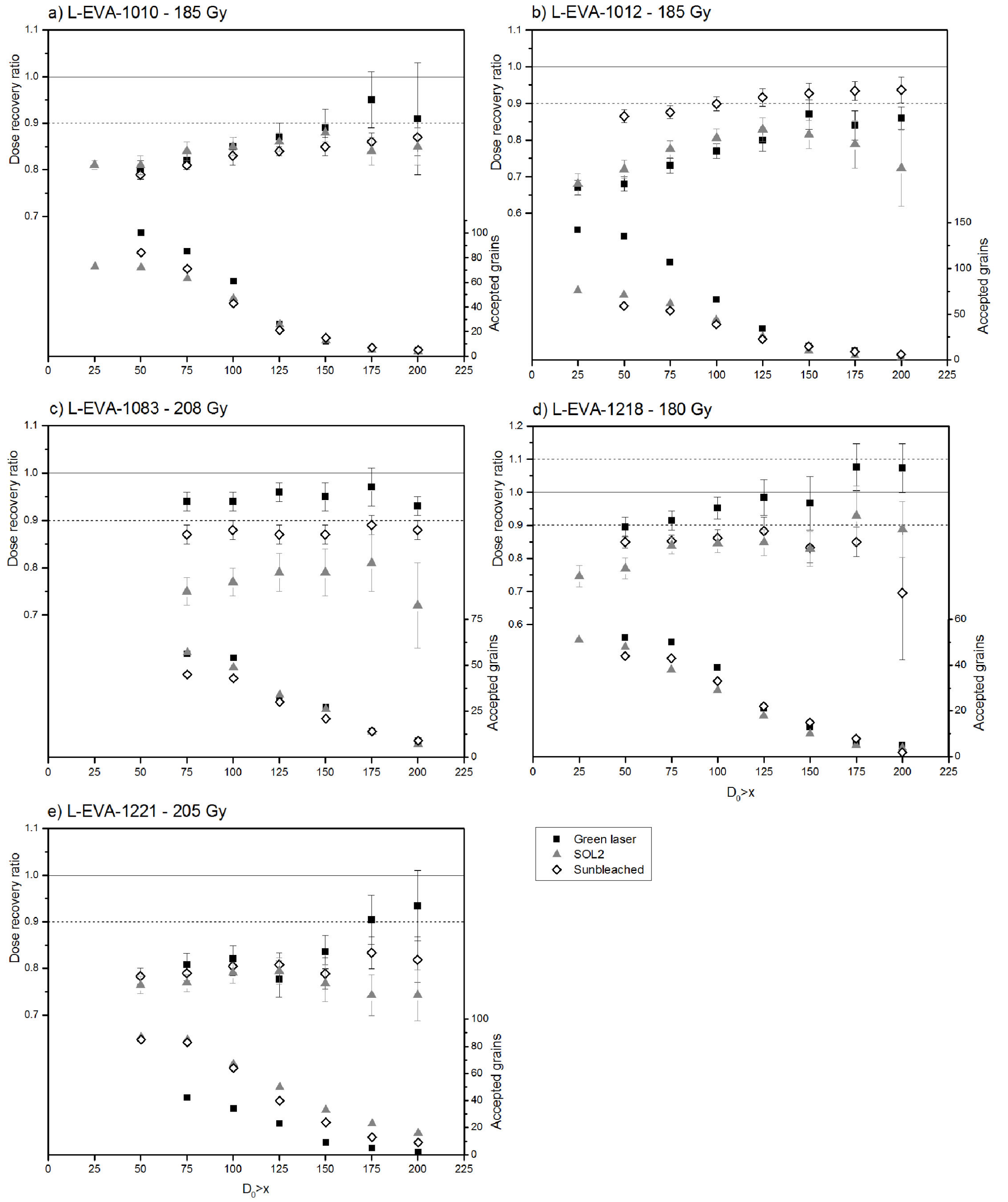

Figure 4. Dose recovery ratio and total amount of accepted grains as a function of single grain D_0 values in dose recovery experiments, with high given doses for each sample in this study.

grain rejection criteria in our dose recovery experiments, we compared dose recovery ratios and σ_{OD} values both before and after application of single grain rejection criteria (Table 1 and Table S3). The dose recovery ratios prior and subsequent to application of rejection criteria are – with the exception of L-EVA-1218 following green laser bleaching and a low given dose – indistinguishable from one another within the given error ranges. σ_{OD} values, however, were reduced considerably (up to 12 %) following application of rejection criteria in 15 out of the 30 dose recovery experiments. We, therefore, argue that rejection of individual grains due to the chosen criteria reduces σ_{OD} values significantly in our experiments.

We further investigated the impact of applying the $D_e > 2D_0$ and the D_e error $< 30\,\%$ criteria in our samples. We undertook this exercise in response to the argument that those criteria are more likely to reject individual grains with high dose estimates rather than low dose estimates, and may consequently bias our single grain dose distributions toward underestimated dose recovery ratios and reduced σ_{OD} values. We calculated dose recovery ratios and σ_{OD} for each experiment in this study using all rejection criteria listed in Section 3, with the exception of $D_e > 2D_0$ and D_e error $< 30\,\%$ (Table S4). Our results from this exercise indicate that application of those two rejection criteria has no significant impact on dose recovery ratios and σ_{OD} values, in 29 out of 30 experiments. Only in sample L-EVA-1083 following green laser bleaching and a high given dose, do dose recovery ratios improve and σ_{OD} is significantly reduced. We then compared dose recovery test results prior to application of rejection criteria (Table S3), with results when all rejection criteria, except the $D_e > 2D_0$ and the D_e error $< 30\,\%$ were applied (Table S4). This comparative exercise shows that σ_{OD} values are still significantly reduced for 14 out of 30 dose recovery experiments, and for only one dose recovery ratio. Our findings suggest that there is no clear justification for the assumption that application of $D_e > 2D_0$ and D_e error $< 30\,\%$ criteria bias single grain dose distributions in our samples. Furthermore, our results also do not indicate that those two rejection criteria cause the apparent reduction of σ_{OD} observed prior and subsequent to application of rejection criteria – at least in the case of the samples analysed in this study.

5.3. Bleaching methods

The impact of different bleaching methods on the dose recovery characteristics of individual grains is almost negligible when given doses are small (Fig. 3, Table 1). With the exception of L-EVA-1083 following sunlight bleaching, all measured/given ratios lie within unity and σ_{OD} ranges from 5 – 11 %. It is, however, interesting to note that a combination of green laser stimulation and low given doses consistently yields the highest proportions of acceptable grains in all samples.

When given doses exceed 180 Gy in the dose recovery experiments, variability in dose recovery ratios and σ_{OD} increases substantially compared to results following low administered doses. No obvious pattern can be observed when comparing results following sunlight and green laser bleaching, leading to the conclusion that there is most likely an additional factor influencing dose recovery results that is best described as a sample-specific response to dose recovery test parameters. This assumption is supported by the fact that both the proportion of accepted single grains, as well as the specific rejection criteria, show dependency on the sampling location.

SOL2 bleaching results – with the exception of L-EVA-1010 – considerably underestimate dose recovery ratios (25 – 32 %) and oversee a substantial increase in σ_{OD} (19 – 35 %) for all samples. In their study, Choi et al. (2009) observed an underestimation of the given dose (10 – 150 Gy) by ∼20 % after SOL2 treatment (> 1 h) as a consequence of sensitisation of the quartz signal during or after the first OSL measurement. Although we chose a relatively short SOL2 stimulation time (5 min) in our experiments, and sample L-EVA-1010 seemed not to be affected at all, we cannot rule out that sensitisation of the material might have taken place either during bleaching treatment or preheating prior to the first OSL signal measurement.

5.4. Administered dose

In our experiments, the most conspicuous factor driving dose recovery characteristics and especially σ_{OD} is the size of the administered dose, as has previously been argued by Thomsen et al. (2012). Determined measured/given ratios and their associated σ_{OD} in three out of five samples are undeniably dependent on the given dose, as is the oversaturation criterion for rejection of single grains.

Thomsen et al. (2016) showed that the dependency of dose recovery ratio and σ_{OD} on the administered dose can be eliminated in dose recovery experiments (with given doses > 30 Gy) by excluding individual grains with D_0 values greater or equal the given dose. They argue that underestimation of dose recovery ratio is related to the inclusion of grains with comparatively small D_0 values, and that by applying the D_0 selection criterion one is likely to discard saturated grains that might otherwise bias dose recovery test results. Following the procedure described in Thomsen et al. (2016), we plotted dose recovery ratios for our experiments with high given doses (> 180 Gy) as a function of $x > D_0$, with x ranging from 0 – 200 Gy (Fig. 4, Table S5). In addition, we show the total amount of accepted grains that pass all rejection criteria for each $x > D_0$. Our observations are similar to those made by Thomsen et al. (2016); dose recovery ratios generally increase with increasing D_0 value. At a threshold value of $D_0 > 125 – 175$ Gy (depending on the sample), however, dose recovery ratios appear to decrease. This is probably linked to the small quantity of individual grains passing D_0 rejection criterion > 125 Gy (Table S5), which may be too few to allow reliable statistical age modelling. Although both dose recovery ratios and σ_{OD} improve in most of our experiments when the D_0 exceeds the given dose criterion is applied (Thomsen et al., 2016), the dose recovery ratios falling closest to unity in most of our samples occurred when $D_0 > 125 – 175$ Gy. This is the case even though not all recovery ratios lie close to unity, as was shown by Thomsen et al. (2016). The total amount of accepted grains from our samples are, however, significantly reduced to fewer than 15 grains (for 10 out of 15 experiments). Thus, we conclude that while rejection of individual grains based on their D_0 values can significantly improve dose recovery ratios and the associated σ_{OD}, it also reduces the quantity of accepted grains overall. This might present problems for statistical age modelling.

6. Conclusion

Our results demonstrate that while the main driving factor influencing beta dose recovery test ratios and σ_{OD} on single quartz grains is the size of the administered dose, sample-specific responses to chosen test parameters (size of the given dose, bleaching type) can also significantly alter the obtained results. High variability in dose recovery test results was observed that is unlikely to be derived solely from the size of the administered dose, particularly for the less sensitized Moroccan samples. Therefore, caution is advised when performing dose recovery tests on samples which are likely to have undergone relatively few sensitization cycles. We conclude that further studies are required to improve our understanding of the range of effects that irradiation time and laboratory bleaching method might have on individual samples.

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